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Influence of surface treatments on fatigue life of Al 7010 alloy

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ABSTRACT

The objective of the present work is to show the influence of machining and anodizing processes on fatigue life of alloy 7010-T7451. Two different cutting conditions were employed to obtain two different initial surface roughnesses. Degreasing, pickling and anodizing were then carried out. In the as machined condition, surface roughness is clearly effective in reducing fatigue life in this material. As the surface roughness increases fatigue life decreases and this effect is found to be more pronounced in high cycle fatigue where major portion of fatigue life is consumed in nucleating the cracks. Effects of pre-treatments, like degreasing and pickling employed prior to anodizing, on fatigue life of the given alloy were also studied. Fatigue curves showed that pickling had negative impact on fatigue life of specimens while degreasing showed no change in fatigue life. The small decrease in fatigue life of anodized specimens as compare to pickled specimens is attributed to brittle nature and micro-cracking of the coating. Scanning electron microscopic (SEM) examination revealed multi-site crack initiation for the pickled and anodized specimens. SEM examination also showed that pickling process attacked the grain boundaries and the inclusions present on the surface resulting in pits formation. These pits are of primary concern with respect to accelerated fatigue crack nucleation and subsequent anodized coating formation.

1. Introduction

Aluminium alloys are extensively used in aeronautical industry to manufacture different structural components due to their superior strength to weight ratios. Fatigue life of a machined component depends strongly on its surface condition. Fatigue cracks are generally considered to nucleate at the surface and therefore surface topography generated by machining plays an important role in determining the fatigue life as demonstrated by Taylor and Clancy (1991) for En19 steel and Wiesner et al. (1991) for Al7075 alloy. The latter performed fatigue tests on cylindrical specimens which have been turned by varying feed rates to produce wide range of surface topography to study its effect on fatigue life. Suraratchai et al. (2008) have established that surface roughness is supposed to introduce stress concentrators that encourage the crack nucleation and accelerates the early fatigue crack growth, hence reducing fatigue life compared to perfectly smooth specimens. Being subjected to different environmental conditions, aluminium alloys are anodized to enhance their resistance against corrosion and wear. Anodizing is well-known electrolytic process that produces controlled columnar growth of amorphous aluminium oxide on the surface of aluminium alloys as documented in ASM Handbook (1998). Despite the benefits obtained by anodizing in terms of corrosion resistance, it has been shown (Cree and Weidmann, 1997) and (ESDU document 87026, 1994) that anodizing has adverse effect on fatigue life of aluminium alloys. It is generally accepted that this reduction in fatigue life is directly attributed to the brittle and porous nature of oxide layer and tensile residual stress induced during anodizing process as demonstrated by Camargo and Voorwald (2007) for alloy 7050-T7451 and Cirik and Genel (2008) for alloy 7075-T6. Prior to anodizing, aluminium alloys undergo pre-treatment solutions comprising of degreasing and pickling, and objective is to produce chemically clean surface ready to be anodized. Localized corrosion, in the form of pits, occur during the pre-treatment solution exposure and these pits has been identified as cause for accelerated crack nucleation during subsequent fatigue loading as documented by Pao et al. (2000) for 7075-T7351 and Dolley and Wei (2000) for 2024-T3. Barter et al. (2002) have observed the influence of microstructure and different surface treatments on the growth of small cracks in a typical high strength aluminium alloy 7050-T7451. They reported that pickling can lead to pitting of grain boundaries with significant fatigue life implication. A recent study by Savas and Earthman (2008) on alloy 7075-T73 showed that various anodizing pre-treatment solutions have different localized corrosion damage. By using SEM they characterized post exposure surface corrosion and put an effort to establish optimum conditions to minimize localized corrosion defects. Abramovici et al. (1991) showed that changing the pickling time had a great influence on fatigue life for 7000 series. Since in the presence of these surface defects, fatigue failure of a compo-

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Table 1
Chemical composition of 7010.

Element	Si	Fe	Cu	Mn	Mg	Cr	Zn	Zr	Ti	Al
Weight%	0.12	0.22	1.98	0.20	2.86	0.15	6.45	0.27	0.14	Bal.

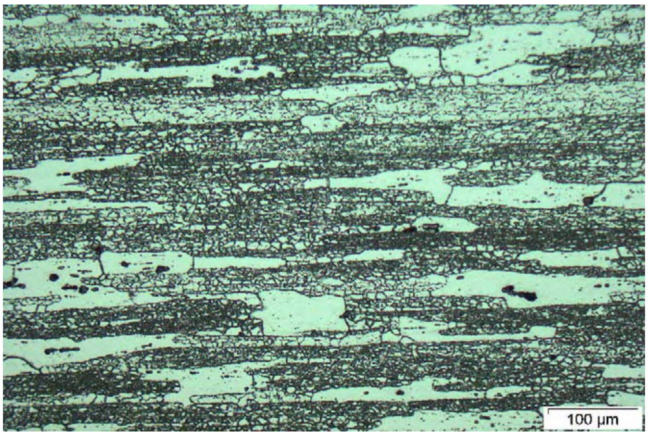

Fig. 1. Micrograph of 7010-T7451 showing recrystallized and unrecrystallized grains.

nent can be greatly accelerated, an inclusive understanding of these pre-treatments on corrosion mechanisms is therefore of scientific interest.

The scope of this work is surface characterization and to demonstrate the coupled effects of surface roughness and pre-treatments, degreasing and pickling, along with anodization on fatigue life of Al 7010 alloy. Since grain boundary orientation along with inclusions distribution and size will be different for longitudinal L, long transverse T and short transverse S directions, thus localized corrosion behaviour will be different for different pre-treatment solutions. However, in this article only LS plane has been studied to investigate different effects of surface treatments on the fatigue life. Scanning electron microscope equipped with energy dispersive spectroscopy (EDS) was used to analyze the fractured surfaces and to identify crack origin sites for specimens with different surface treatments.

2. Experimental procedures

2.1. Material

The investigated material in this work is 7010-T7451 whose chemical composition as determined by EDS of mark *Quantax* is given in Table 1.

The material was provided in rolled plate form of 70 mm thickness. T7451 treatment consists of heat-treating, quenching and overaging (Lyman, 1967) for improved fracture toughness and minimal loss of tensile strength. Metallographic analysis of the microstructure revealed that it is composed of unrecrystallized and recrystallized grains and latter are highly elongated in the rolling direction as shown in Fig. 1. Mainly two types of inclusions were found in this material: Mg_2Si, Al_7Cu_2Fe and these were generally located in recrystallized grains. The average size of these particles varied between 8 and 10 μm.

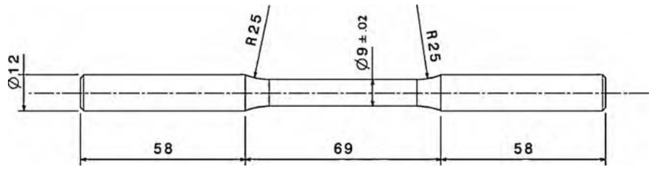

Fig. 2. Fatigue specimen geometry (dimensions in mm).

Table 3
Classification of specimens according to surface treatments.

	Degreasing	Pickling	Anodizing
Group 1	No	No	No
Group 2	Yes	No	No
Group 3	Yes	Yes	No
Group 4	Yes	Yes	Yes

Mechanical properties of the alloy determined by tensile tests are; yield strength 464 and 458 MPa, ultimate tensile strength 526 and 525 MPa and elongation 9.8% and 8.4% in the longitudinal (L) and transverse (T) directions, respectively.

2.2. Specimen preparation

Cylindrical test specimens, as shown in Fig. 2, have been machined by turning without using lubricant. Turning was performed on 2 axes numerical lathe RTN20 of mark RAMO. Test specimens were prepared in such a way that maximum load is applied perpendicular to the rolling direction.

To characterize roughness of a machined surface, various geometric parameters such as average roughness (R_a), peak to valley height roughness (R_y) and 10-points roughness (R_z) are generally used. These parameters are calculated from profile height (z) distribution over an assessment length l. In this study R_a has been used as principal parameter to define surface roughness given as,

$$R_a = \frac{1}{l}\int_0^l |z(x)|dx$$

Two types of machined surfaces ($R_a = 0.6$ μm and $R_a = 3.2$ μm) were produced by varying the feed rate and tool nose radius. The cutting conditions used at lathe for this study are given in Table 2. To cater the tool wear, for each specimen new cutting edge of tool insert was used to avoid large variation in surface roughness.

To illustrate the different surface treatment effects on fatigue life, specimens were categorized in 4 groups as shown in Table 3.

The first group, involving low and high roughness specimens, with no surface treatments was used to build reference fatigue curve for comparison purpose with other three groups.

2.3. Surface treatments

Anodizing is a classical method used to improve the corrosion resistance of aluminium alloys. It is accomplished by making the workpiece anode while suspended in a suitable electrolytic

Table 2
Cutting conditions employed for specimens surface preparation.

Machining parameters			
Feed rate (mm/rev)	Nose radius (mm)	Cutting speed (m/min)	Surface roughness
0.1	0.8	180	$R_a = 0.6$ μm ± 0.1
0.2	0.4	180	$R_a = 3.2$ μm ± 0.15

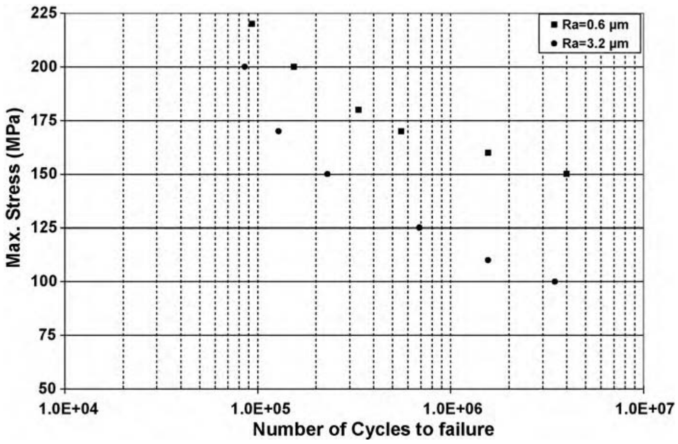

Fig. 3. Fatigue test results for two different surface roughnesses.

cell at suitable temperature and voltage. Before anodizing process, aluminium alloys are subjected to different surface treatments which consist of degreasing and pickling and purpose of these pretreatments is to produce a chemically clean surface. For degreasing, the specimens are immersed in a special detergent that removes oils, grease and solid dust particles from metal surface. Pickling is employed to remove the natural oxides and other compounds from surface by means of a solution which acts chemically upon the compounds. Removal of oxide layer allows for a more conductive surface thus facilitating electrochemical processes such as anodizing.

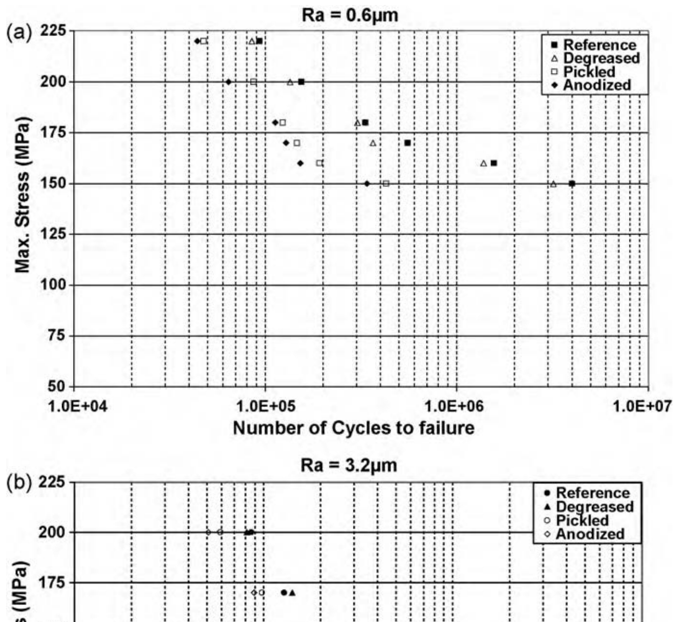

Fig. 4. (a) S–N curves for lower roughness and surface treated specimens and (b) S–N curves for higher roughness and surface treated specimens .

Specimens degreasing was carried out in aqueous solution of sodium tripolyphosphate $Na_2P_3O_4$ and Borax $Na_2B_4O_7$ at 60 °C for 20 min ± 1 min followed by demineralised water rinsing. Pickling was done in aqueous solution of H_2SO_4 acid and anhydride chrome CrO_3 at 60 °C for 8 min ± 30 s followed by rinsing. Chromic acid anodization was accomplished in anhydride chrome CrO_3 solution at 45 °C under 50 V for 55 min ± 1 min. The average thickness of oxide layer produced by the process was measured to be about 3 µm. The solution baths were normally agitated during surface treatments.

2.4. Surface measurement

Surface roughness profiles were obtained using Mahr PKG120 profilometer. It is a diamond stylus instrument that can give conventional roughness parameters with horizontal and vertical resolutions of 0.5 and 0.1 µm, respectively. For each specimen four readings were taken with measuring length of 5.6 mm each and then average was calculated. Roughness measurements were performed after each surface treatment to see its effects. There was slight increase in surface roughness 'R_a' parameter after pickling which is due to the formation of pits.

2.5. Fatigue testing

Rotating bending fatigue tests were performed at frequency of 60 Hz in laboratory conditions to obtain the S–N curves. The choice of this type of testing was made because maximum stress is applied to the specimen surface and by the nature of rotating bending test, the stress ratio R was −1.

3. Results and discussions

3.1. S–N curves

The result of rotating bending fatigue tests for first group is shown in Fig. 3. Surface roughness is clearly effective in reducing fatigue life for this material. In low cycle regime there is about 10% decrease in fatigue strength while in high cycle regime there is 32% decrease. This confirms that surface roughness plays a vital role especially in high cycle fatigue in which major part of fatigue life is consumed in nucleating the cracks.

S–N curves for the specimens with low and high roughness after surface treatments i.e. degreasing, pickling and anodizing are shown in Fig. 4(a) and (b), respectively. The degreasing showed no remarkable influence on fatigue life, for both surface conditions, while pickling was found damaging in reducing fatigue life con-

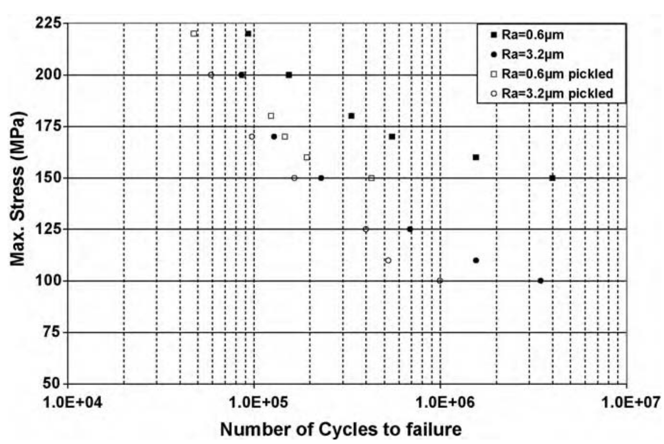

Fig. 5. S–N curves for machined and pickled specimens.

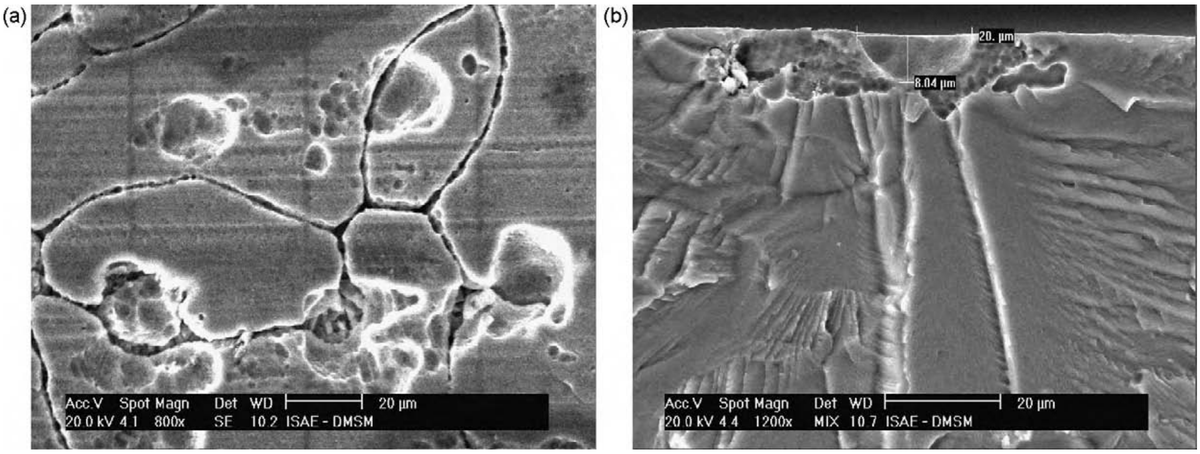

Fig. 6. (a) Pickling process resulting in the pit and (b) fatigue crack initiated from one of pit formation at grain boundaries at the surface.

siderably. This decrease in fatigue life caused by pickling could be associated with degradation of surface condition as compared to machined surface of the specimen. The presence of pits on the specimen surface plays a vital role in accelerating the fatigue crack nucleation and their subsequent growth.

From graphs it is also clear that anodization decreased the fatigue life slightly as compare to pickled specimens for this alloy. This decrease could be attributed to the brittle nature of the oxide coating, which readily crack when loaded, and due to the presence of micro-cracks in the coating, which may result in an early fatigue crack initiation. Since oxide layer adheres extremely well to substrate, any crack that develops in it acts like stress raiser and propagates to substrate. Another important aspect is that pickling effect is more prominent for low roughness specimens than higher roughness specimens especially in high cycle fatigue as can be seen in Fig. 5. Analysis of these S–N curves using Basquin's model allows in defining the loss of fatigue life. For the lower roughness specimens, loss is 32.6% for 10^6 cycles as it is only of 12.4% for 10^5 cycles. For higher roughness specimens, loss is only of 18.7% at 10^6 cycles and 7.7% for 10^5 cycles. Through these results, it appears that the effect of pickling is 50% more important in case of initial lower roughness.

3.2. SEM observations

The fractured surfaces of the specimens with different treatments were examined by SEM to identify the crack origin sites. In addition, EDS was used for inclusions identification for those found to nucleate the cracks. For the specimens in as machined condition, fatigue cracks had been observed to nucleate at inclusions Mg_2Si and Al_7Cu_2Fe present at the surface. In some cases, inclusions are not exactly at surface but are very close to it, usually at 10 µm at its closest point. Patton et al. (1998) studied the same alloy and also showed that most of cracks initiated by the fracture of inclusions present in this alloy. Pickling process was found to attack the grain boundaries and inclusions resulting in pits formation on the surface as shown in Fig. 6(a) and (b), which in turn acted as stress concentration facilitating crack initiation and also promoting crack growth. The pickling pits which were found to nucleate fatigue cracks were about 8 µm deep. In some cases, where two pits were close to each other, cracks nucleation grew together to form single crack front. Presence of the many pits on the surface also explains multi-crack initiation sites for the specimens which were treated with pickling solution as shown in Fig. 7 as compared to machined specimens. This phenomenon of multiple crack nucleation was observed for both initial surface roughnesses, i.e. R_a = 0.6 µm and R_a = 3.2 µm for group 3 specimens.

It was revealed by SEM observation that some inclusions were attacked on their periphery and some were dissolved to various extents leaving a trace behind as shown in Fig. 8. This phenomenon is also reported by Biribilis and Buchheit (2005); they showed that inclusion type had an influence on pitting process and classified pit morphologies into two categories for aluminium alloys. One type is designated as circumferential that appears a ring of attack around an inclusion and other type is referred to as selective dissolution. Pits structures for latter type are typically deeper and may have

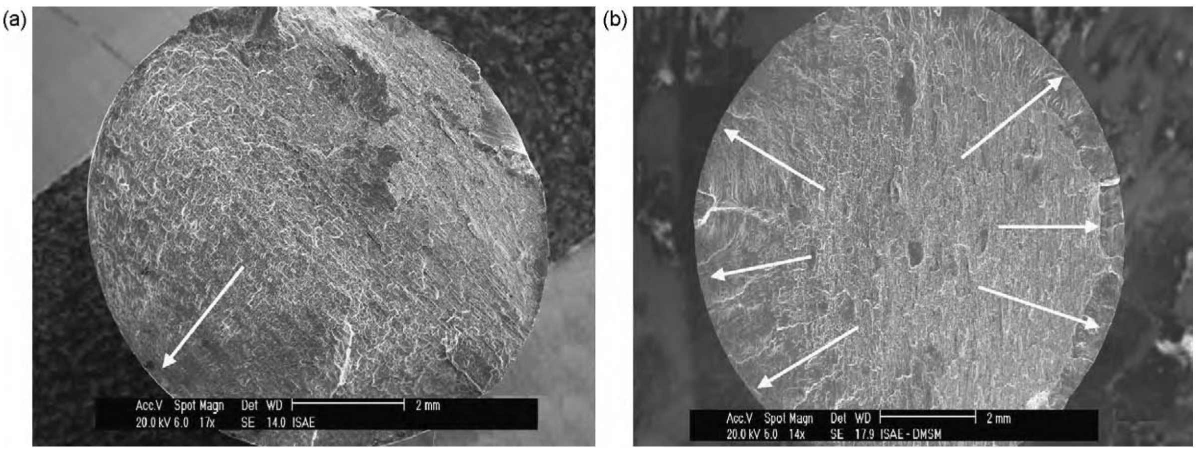

Fig. 7. (a) Single crack nucleation site for specimen in as machined condition and (b) multi-crack nucleation sites for specimens treated with pickling solution.

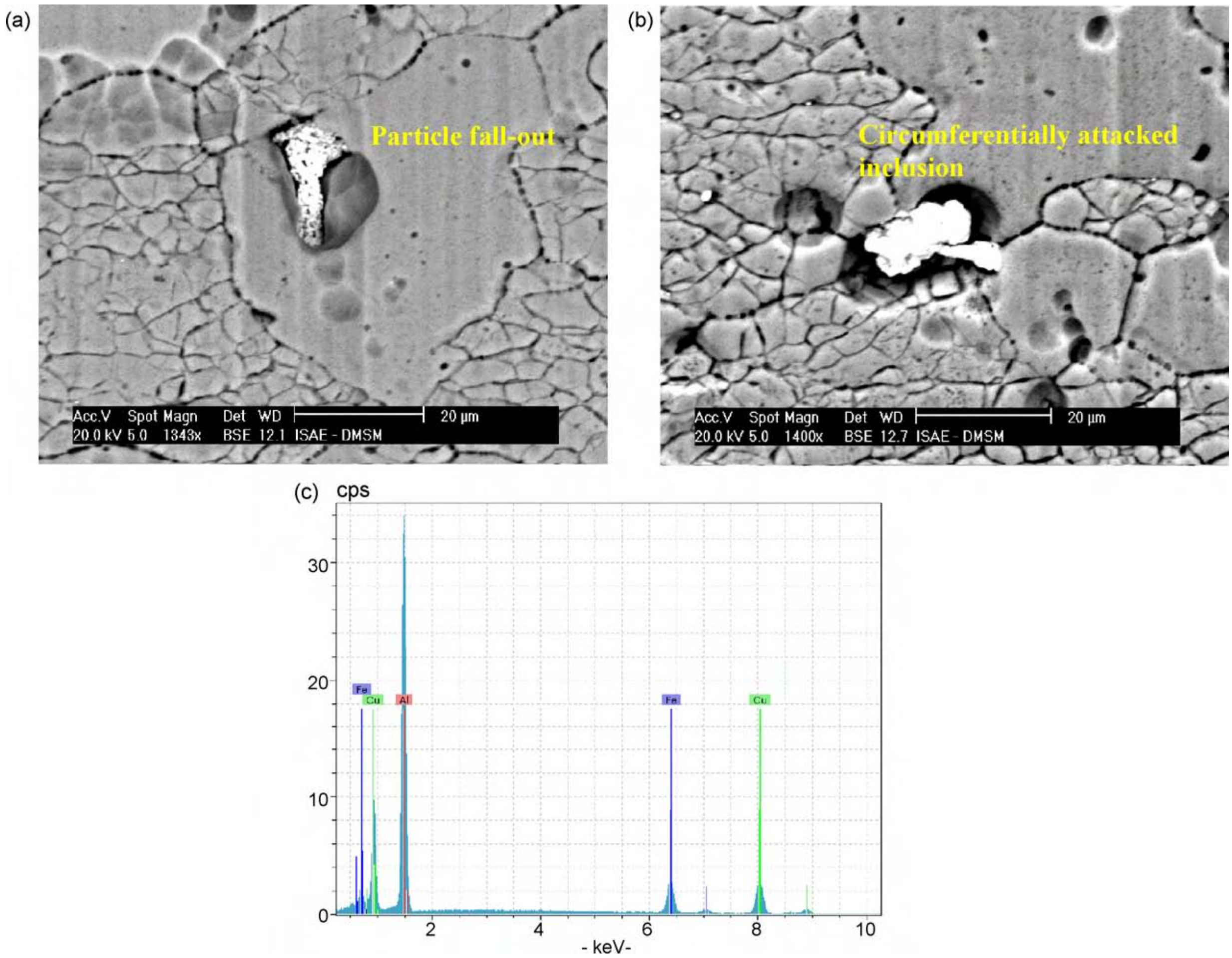

Fig. 8. (a) Particle fall out and circumferentially attacked inclusion (b) circumferentially dissolved inclusion and (c) EDS spectra for Al_7Cu_2Fe.

remnants of particle in them. This type of damage has also been referred to as particle fall out. EDS analyses were conducted on these pits, Fig. 8(c), to identify the type of inclusions attacked by the pickling solution and found that mainly it was Al_7Cu_2Fe.

With the help of a profilometer, topography measurements of pickled specimens were carried out to show the presence of these pits like defects. For the purpose of clarity only one profile extracted from topography is presented and down peaks in Fig. 9 correspond to the pits.

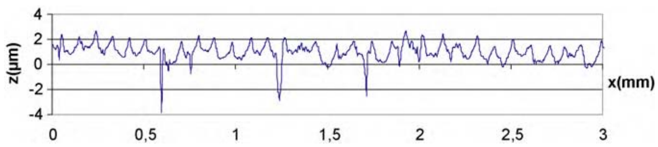

Fig. 9. Profile obtained by topography.

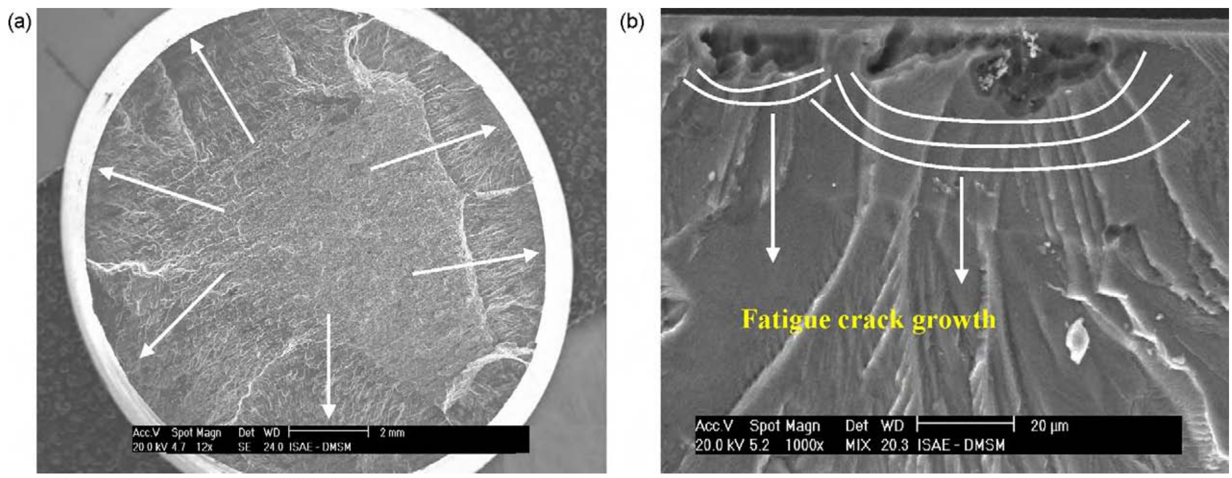

Fig. 10. (a) Specimen with anodized coating with and (b) fatigue crack started from pits beneath multi-site crack nucleation the anodized coating .

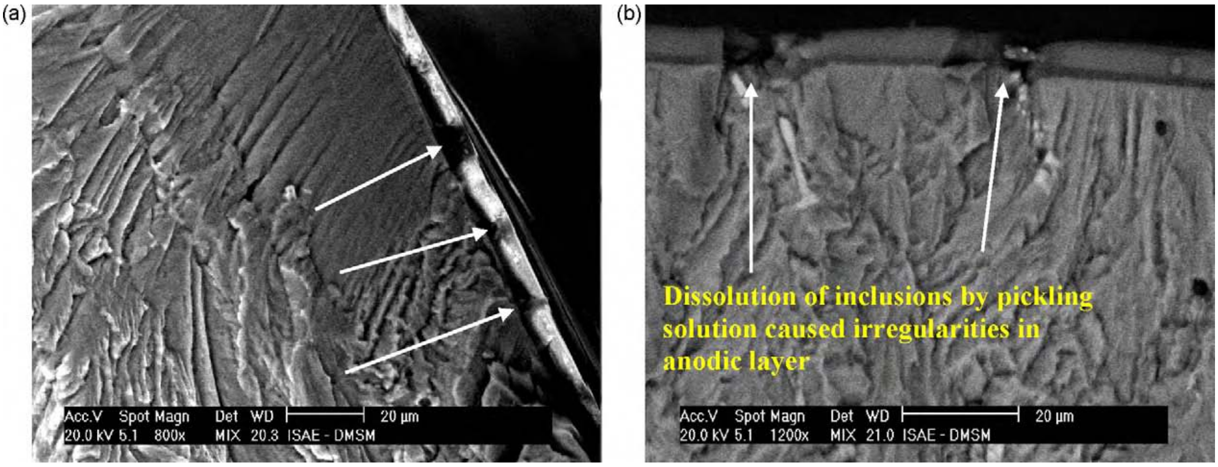

Fig. 11. (a) Partial delamination of coating from substrate and (b) irregularities found in the coating.

It is evident from Fig. 10 that multiple crack nucleation sites were also found on the fractured surface of the anodized specimens. Shiozawa et al. (2001) also demonstrated this phenomenon that number of crack initiation sites increased for the anodized specimens compared to untreated ones. When analyzed thoroughly by SEM, it was found that most of the fatigue cracks for anodized specimens were initiated by pickling pits beneath the coating as shown in Fig. 10(b) while others were started from coating and the propagated to substrate. For relatively small pits, it was discovered that anodic coating was actually able to grow over these pits and produce relatively a smoother surface.

Although anodized coating was found to be quite uniform in most regions around the specimen, SEM examination showed that in some region there was partial delamination; Fig. 11(a) indicated by arrows, at the interface of coating and substrate and this delamination may be related to the interfacial shear strength as reported by Cirik and Genel (2008). Also the presence of irregularities (Fig. 10(b)) beneath the coating is important consideration affecting crack nucleation and propagation. These irregularities can act like local stress concentrations and can result in delamination and cracking of the coating. The presence of these irregularities in the coating can be correlated to the presence of pickling pits and inclusions as shown by Fig. 10(b).

4. Conclusions

Based on the results obtained by rotating bending fatigue tests on alloy 7010-T7451 and SEM observations, the following conclusions are made:

i. Degreasing showed no change in fatigue life but pickling was found to be detrimental in reducing the fatigue life significantly. It was shown by SEM observations that pickling attacked the grain boundaries and inclusions present on surface resulting in pits formation and these pits acted like stress raisers. SEM observations also revealed multi-crack initiation sites for pickled specimens for both surface roughnesses.
ii. Fatigue life of the given alloy is highly dependant on its surface topography. As the surface roughness increases fatigue life decreases and this reduction in life is more prominent in HCF where major part of fatigue life is consumed in nucleating the cracks.
iii. Anodization slightly reduces the fatigue life for both surface conditions in this alloy as compare to pickling. The brittle nature of anodized coating and irregularities beneath the coating are the factors for this small degradation. These irregularities can be associated to pits formed during pickling and inclusions present on the surface.
iv. By comparing the fatigue decrease for pickled and anodized specimens, this may be concluded that decrease caused by pickling process is more than anodized ones. This suggests that pickling pits significantly influence the fatigue behaviour than anodized coating for the given alloy and that a compromise has to be found between machining conditions for surface roughness and anodizing parameters, specially pickling according to the best value cost/fatigue performances.

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